4 FIRST TOPICAL REPORT 6

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3 STABILITY OF STRUCTURAL MATERIALS FOR SPACECRAFT APPLICATION 4

to

GODDARD SPACE FLIGHT CENTER
Greenbelt, Maryland

Contract No. NAS 5-10267 29 CY

9 December 30, 1966 10 CY

by

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SUMMARY

This program has been designed to evaluate some of the material parameters related to the dimensional stability of various materials. In particular, measurements have been made of the microyield stress (MYS, the stress associated with an offset yield of 1×10^{-6}) for aluminum alloys 2024, 5456, and 6061, I-400 beryllium, TZM molybdenum, and AZ 31 magnesium. These data, along with a description of the test methods, are presented.

INTRODUCTION

It is necessary that the dimensions of many spacecraft components be under very rigid control during ground handling, launch, and operational conditions. That is, distortions due to thermal gradients, environmental stress, or inherent dimensional instabilities must be held to an absolute minimum. One can calculate quite well, on the basis of known thermal expansion, thermal conductivity, heat capacity, etc., the response of a structure to an assumed thermal environment. However, for many of the materials of interest, the data required to estimate the effect of an assumed stress environment are simply not available. It is the purpose of this report to describe the initial yield behavior of some materials so as to provide some information as to the maximum short-time stress to which such materials might be exposed without serious permanent dimensional changes. These data comprise only a part of those being generated on a more comprehensive program. They are intended to provide the baseline data from which suitable creep stresses can be chosen for subsequent experimentation.

In particular, the measurements reported herein are concerned with stress-microstrain relationships (through the first 10 to 50 micro-inches per inch strain). The primary concern is the measurement of the microyield stress (MYS) associated with a permanent deformation of 1×10^{-6} inches per inch. In the past, this has been called the precision elastic limit (PEL), but this term is a misnomer, for it is in no sense an "elastic limit".

One must approach the use of such MYS data with considerable caution. To emphasize this, it is perhaps useful to emphasize the significance of a sensitivity of one part in 10^7, which is approximately the sensitivity of the testing procedures used here. This is the equivalent of measuring a mile to better than 0.01 inch, or to measuring the distance between New York and San Francisco to within 18 inches. When such sensitivities are employed, one must expect that the data can be influenced by a variety of subtleties, not all of which are known and appreciated.

Therefore, one <u>must expect</u> the MYS of duplicate specimens to differ by more than the 0.2 percent offset yield differs. One <u>must expect</u> small differences in surface treatment, composition, deformation, etc., to show up as large differences in MYS. One must also expect, and this is largely borne out by the data, that MYS values can differ greatly from conventional elastic-limit or offset-yield values.

MATERIALS AND MATERIAL PREPARATION

The materials obtained for the program are characterized by composition, source, and condition, as shown in Tables 1 and 2, respectively.

Secondary heat treatments given for stress relief are shown in Table 3.

Tensile specimens of the design shown in Figure 1 were prepared from each of the materials. To reduce machining stresses and to remove any remaining effects of the machined surface and of the subsequently heat-treated surface, about 10 mils were removed from the specimen thickness in the necked portion of the specimen by chemical milling. Chemical solutions and milling temperature for each alloy are listed as follows:

(1) <u>Aluminum Alloys</u>

Sodium hydroxide (NaOH)	100 g
Water	500 cc
Bath temperature	~110 F

Remove smut in 10 percent HNO_3 in water.

(2) <u>Beryllium I-400</u>

Sulphuric acid (96% H_2SO_4)	27 cc
Phosphoric acid (85% H_3PO_4)	410 cc
Chromic acid (CrO_3)	70 g
Water	130 cc
Bath temperature	~170 F

TABLE 1A. MATERIAL COMPOSITION

	Al	C	Cu	Co	Fe	Mg	Mn	Si	Zn	Be	BeO
Aluminum 2024	Bal	--	4.5	<0.01	0.26	1.42	0.62	0.22	<0.2	--	--
Aluminum 5456	Bal	--	<0.01	0.20	0.18	2.30	<0.01	0.09	<0.2	--	--
Aluminum 6061	Bal	--	0.25	0.26	0.52	0.76	0.07	0.60	<0.2	--	--
Beryllium	0.07	0.20	--	--	0.13	0.01	0.01	0.04	--	Bal	5.78

TABLE 1B. MATERIAL COMPOSITION

	Al	C	Fe	Mn	Ti	Zn	Zr	O	H	N	Mo	Mg
Molybdenum TZM	--	0.016	<0.001	--	0.42	--	0.087	<0.0003	<0.0001	<0.0003	Bal	Bal
Magnesium AZ 31	3.2	--	--	0.43	--	1.05	<0.1	--	--	--	--	Bal

TABLE 2. MATERIAL CONDITION, QUANTITY, SIZE, AND SOURCE

Material	As-Received Condition	Quantity	Size, inches*	Source
Aluminum				
2024	T4	30 strips	1/8 x 1 x 4	Williams and Company, Columbus, Ohio
5456	H34	30 strips	1/4 x 1 x 4	Williams and Company, Columbus, Ohio
6061	T6	30 strips	1/8 x 1 x 1	Williams and Company, Columbus, Ohio
Beryllium I-400	Hot pressed	1 block	1 x 4 x 2.5	Brush Beryllium Company, Cleveland, Ohio
Molybdenum Mo-0.5Ti	Stress relieved	24 strips	1/8 x 1 x 4	Climax Molybdenum Company of Michigan, Ann Arbor, Michigan
Titanium				
6Al-4V	Solution treated and aged	27 strips	1/8 x 1 x 4	Titanium Metals Corporation,of America, Cleveland, Ohio
5Al-2.5Sn	Annealed	27 strips	1/8 x 1 x 4	Titanium Metals Corporation of America, Cleveland, Ohio
Magnesium AZ 31		2 plates	6 x 6 x 6 14 x 14 x 0.5	Goddard Space Flight Center, Greenbelt, Maryland

*All saw cut with 4-inch length parallel to rolling direction.

TABLE 3. SECONDARY HEAT TREATMENT

	Secondary Heat Treatment			
Alloys	Temperature, F	Time, hours	Cooling	Atmosphere
Aluminum				
2024	400	1	FC[a]	Air
	450	1	FC	Air
	500	1	FC	Air
5456	400	1	FC	Air
	450	1	FC	Air
	500	1	FC	Air
6061	400	1	FC	Air
	450	1	FC	Air
	500	1	FC	Air
Beryllium				
I-400	1100	1	FC	Vacuum
	1500	1	FC	Vacuum
Molybdenum				
Mo-0.5Ti	2200	1	FC	Hydrogen
	2600	1	FC	Hydrogen
Magnesium				
AZ 31	450	1	FC	Air

(a) FC denotes furnace cooling. Cooling rate not to exceed 300 F/hr from 2600, 200 F/hr from 1600 to 500 F, and 100 F/hr from 500 F to room temperature.

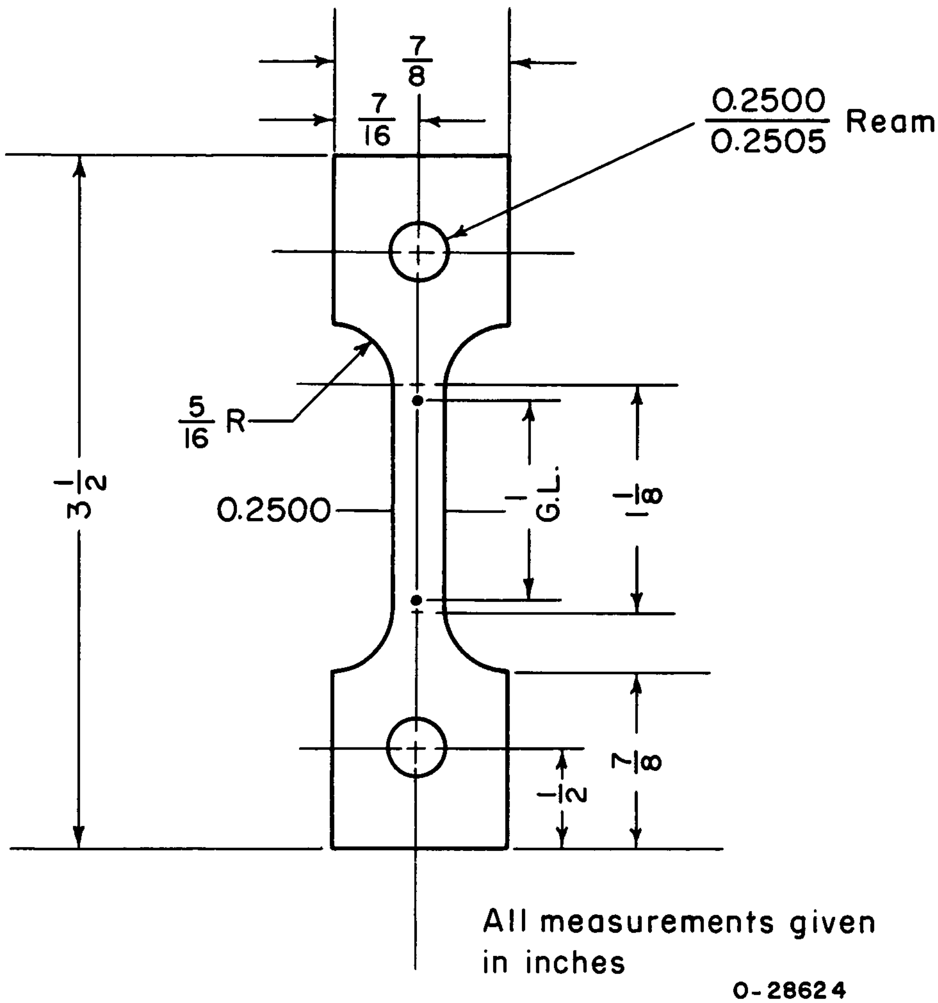

FIGURE 1. SPECIMEN FOR TENSILE MYS TESTS

Note: Holes to be located on axis of reduced section to within 0.0005 inch. Thickness of specimen to be 0.1220 or less.

(3) Molybdenum TZM

Water	225 cc
Nitric acid (70% HNO_3)	150 cc
Sulphuric acid (96% H_2SO_4)	150 cc

(4) Titanium Alloys

Lactic acid (88.4% $CH_3CHOHCOOH$)	240 cc
Nitric acid (70% HNO_3)	80 cc
Hydrofluoric acid (48.7 HF)	80 cc

Slowly stirred on a magnetic stirrer

(5) Magnesium AZ 31

5 percent nitric acid in water

Chemical milling was accomplished by the following procedure. Stop-off materials (such as Microstop lacquer, which was applied by brushing or dipping, and microwax, which melts at 125 F and was applied by dipping) were used to mask the heads of the specimens. The masked specimens were placed in the constantly agitated milling bath for various periods of time. The specimens were periodically removed from the bath, washed, and measured. Complete milling cycles ranged from 10 to 30 minutes for each specimen. Removal of Microstop was accomplished by washing with acetone. Microwax was removed by cutting the wax off with a razor blade, and removing the residue with trichlorethylene.

Foil gages of Type MA-XX-250 BG-120, manufactured by the W. T. Bean Company, were used throughout this study. Some of the details of these gages are given in Figure 2.

Two types of cements were employed for MYS tests; Eastman 910, a cyanoacrylate cement, and BR-600, an epoxy cement. Eastman 910 is convenient for application, and cures at relatively low temperature. BR-600 requires an elevated-temperature cure to achieve the desired degree of stability and strength.

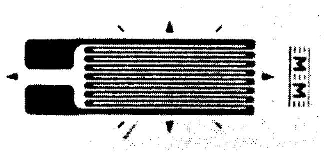

FIGURE 2. MA-XX-250BG-120
FOIL GAGE

Physical Properties:
Dimensions
Gage length 0.250 inch
Overall length 0.375 inch
Grid width 0.125 inch
Overall width 0.125 inch

Grid Alloy
Constantan foil with a temperature compensation for 3, 6, 13, or 15 μin./in./F

Backing
A tough epoxy resin film with a 0.0009-inch thickness

Electrical Properties
Gage resistance 120 ohms
Gage factor 2.095
Linearity better than 0.05 percent

Eastman 910 was used for MYS tests on 5456-H34 Al, 6061-T6 Al, I-400 beryllium, molybdenum TZM, and magnesium AZ 31; BR600 was used for 2024-T4 Al.

With Eastman 910, the chemically milled surface of the specimen was cleaned with alcohol, metal conditioner, and neutralizer. A terminal strip (GS-3 from Budd Company) was placed about 1/16 inch from the tab end of the gage. A piece of cellophane tape (3/4 inch wide and 1.5 to 2.5 inches long) was placed over the open face of the gage and terminal strip for handling. The gage and terminal-strip backings were cleaned with a cotton swab or Q-tip slightly moistened with the neutralizer. A thin film of accelerator was then applied, and one to two minutes drying time was allowed. The tape was oriented on the specimen in the area for gaging. One end of the tape was then attached to the specimen. One drop of cement was placed on the specimen near the end of the gage, and another drop in the middle of the gage area. With some pressure from the thumb, one wiping motion was made from the taped end to the terminal-strip end. The gage was held with moderate pressure for two to four minutes. Then the tape was peeled off at 90 degrees to the gage plane, the specimens were placed in an oven at 105 F, and allowed to post-cure for 24 hours. A protective coating, designated as Gagekote No. 1 by W. T. Bean Company, was applied over the gage, except for the gage tab and terminal strip, as soon as the specimen had been cured. Small soldered flexible leads connect the gage to the terminal strips. The main leads were soldered to the terminal strips. The resin of the solder was dissolved with solvent prior to final and overall coating of the gage and the lead wires. The gaged specimens were ready for testing after drying the coating for one hour or longer.

With BR-600, surface preparation of the specimen was the same as above. The GS-3 terminal strips were again placed on the Mylar tape, 1/8 inch from the tab of the gage. With a neutralizer-moistened Q-tip both faces of the gage and the terminal-strip backing were cleaned and allowed to dry. A thin film of BR-600 was applied to the surfaces of the gage and the terminal-strip backing, and allowed to dry for 5 minutes in air. The gage was then oriented in the designated area and held on the specimen by tape. A 2-inch-long by 3/4-inch-wide 1-mil-thick Teflon sheet was held down on the tape end. With some pressure from the thumb, one wiping motion was made from the tape end to the end of the gage. Similarly, a gage on the opposite face of the specimen was applied. Then with a silicone rubber pad, 1-1/2 inches long, 3/4 inch wide, and 1/8 inch thick on each side of the specimen, the gages were sandwiched with Teflon to protect them from sticking. Aluminum pressure distributors of equal size were placed on the pads and a clamp force of 12 to 60 psi was applied with a small C-clamp. The whole assembly was placed in an oven to be cured at 350 F for 5 hours, followed by furnace cooling. The application of the protective coating and the installation of lead wires were performed in the same manner as above. An example of a prepared specimen is shown in Figure 3.

TESTING PROCEDURES

The system devised to load the specimens in uniaxial tension is shown in Figures 4 and 5. The details of the assembly can be seen in the drawing of Figure 6. The uppermost fitting (in the exploded view) is a ball-thrust-bearing system to eliminate the possibility of significant

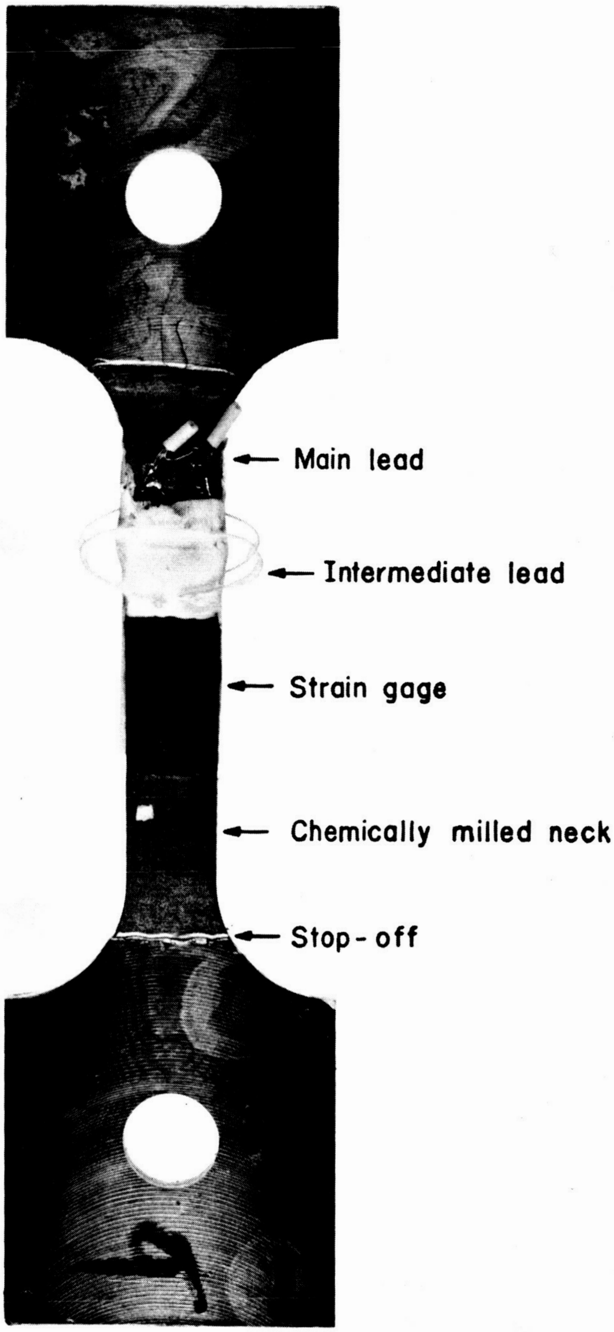

FIGURE 3. TYPICAL SPECIMEN SHOWING STRAIN GAGE DETAILS

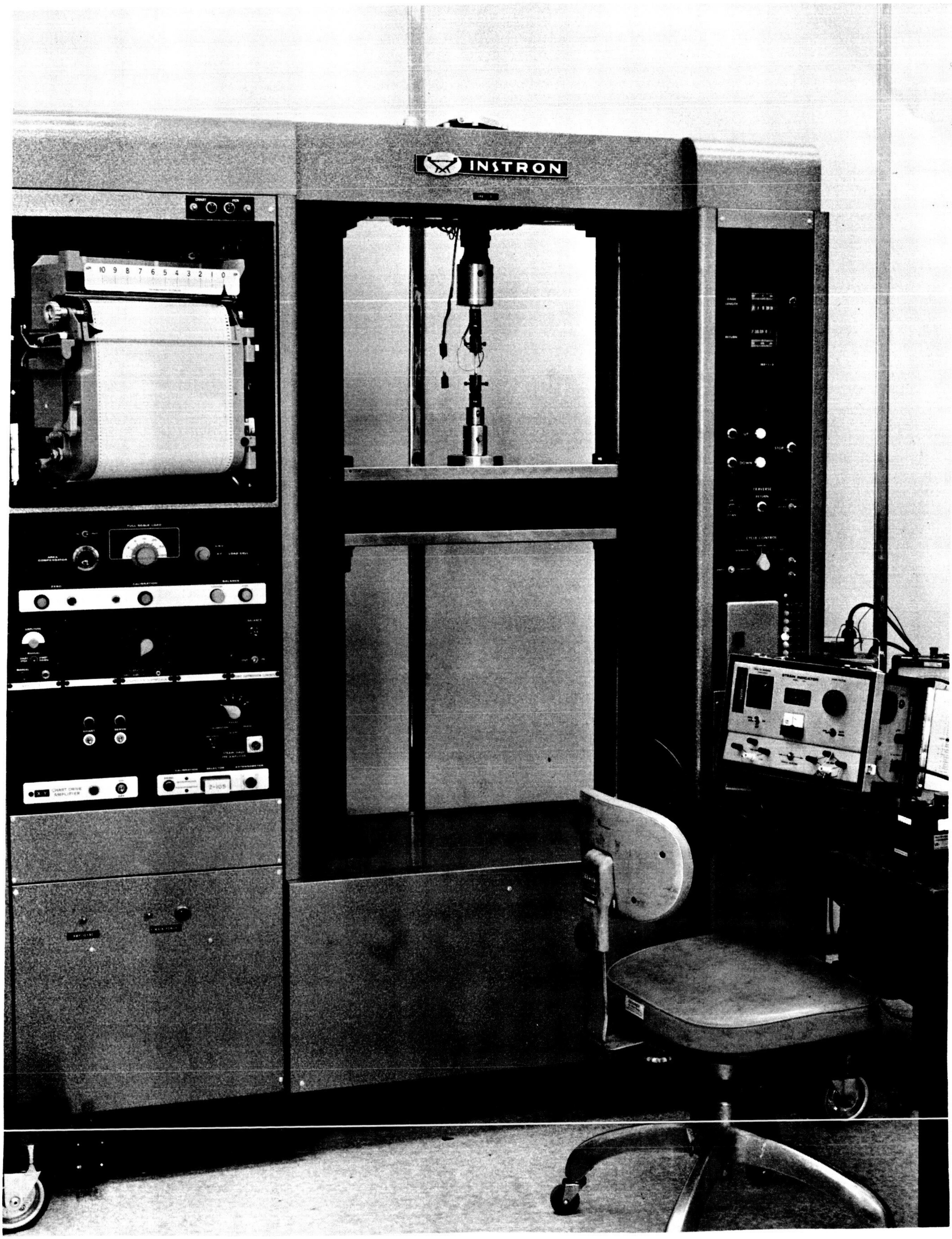

FIGURE 4. MYS TEST SET-UP

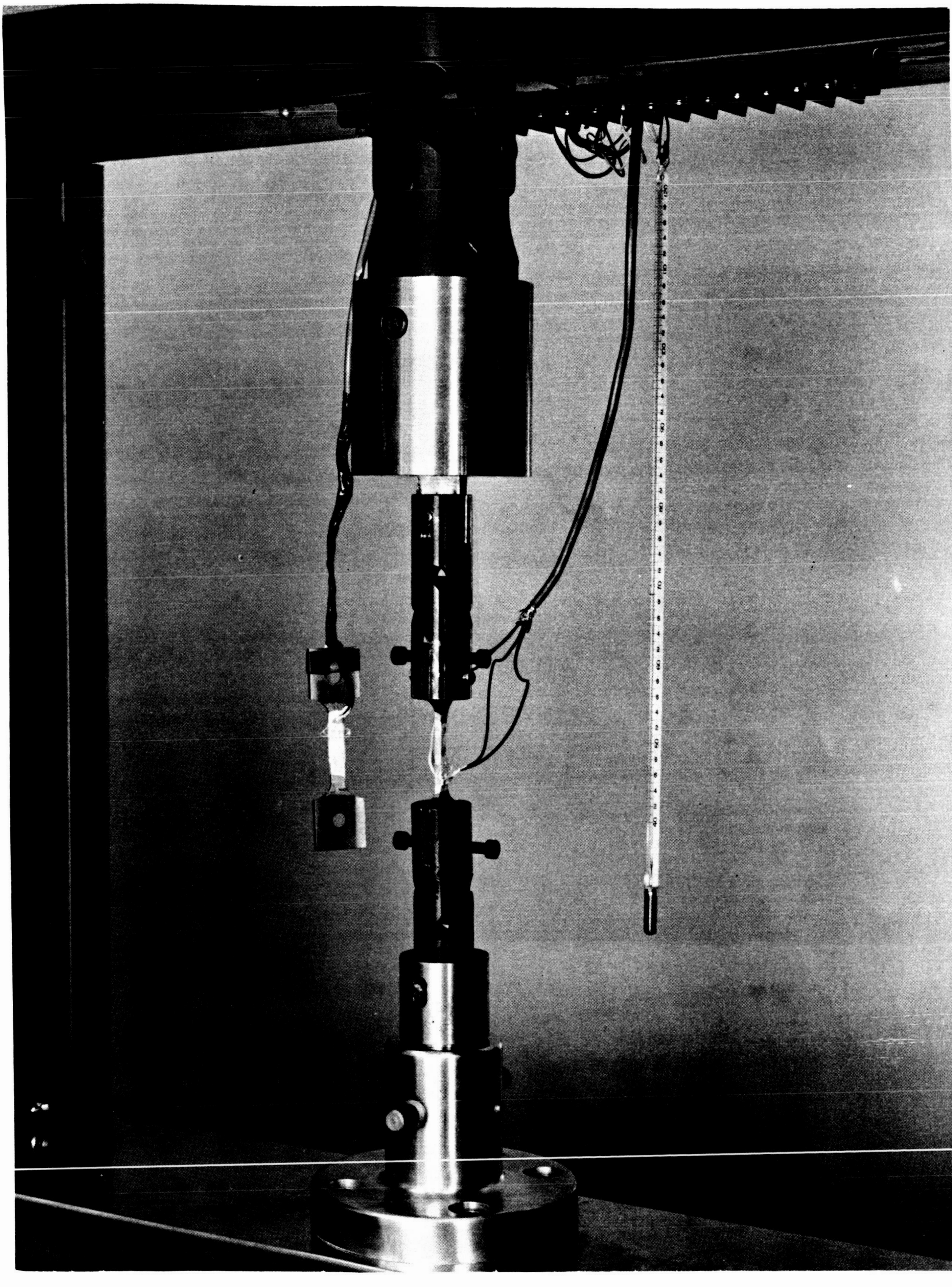

FIGURE 5. CLOSE-UP VIEW OF MYS LOAD TRAIN

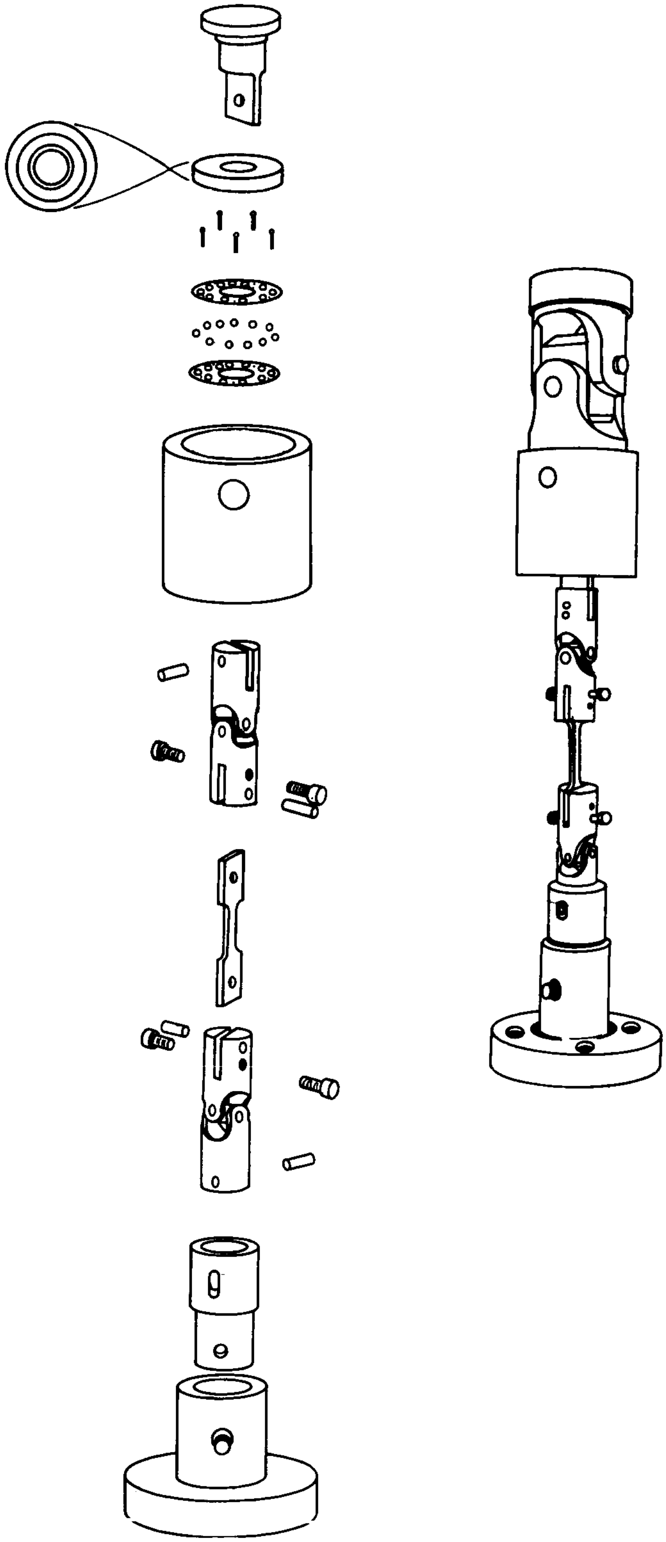

FIGURE 6. EXPLODED VIEW OF MYS LOAD TRAIN

torque being applied to the specimen during loading. This device supports a universal joint in which the specimen is gripped. The lower end of the specimen is gripped in another universal joint attached to the moving platen of the tensile machine by a pinned joint. In this lower joint, the pin is confined in a slotted hole. Therefore, when the tensile load is removed, there is no compressive load applied to the specimen.

Active and dummy gages are connected in full-bridge circuit with Number 25 AWG four conductor wire, shielded in pairs. Bridge connections were made to a terminal strip attached rigidly to the loading frame as shown in Figure 5. Strain measurements were made with a BLH Model 120 strain indicator, factory modified to increase its sensitivity by a factor of 4. The indicator was modified still further by using a highly sensitive galvanometer in place of the normal null indicator.

A full-bridge circuit is used (as shown in Figure 7) with the inactive bridge resistors being gages of the same type and mounted on the same material as the active gages. All the gages were kept close together physically to minimize temperature variations. The use of temperature-compensated gages also aids in minimizing the effects of temperature fluctuations. All tests were conducted in a constant-temperature room ($68 \pm 1/4$ F).

In practice, the specimen is loaded several times up to 5 to 10 percent of its estimated MYS. Both gages are read individually in a half-bridge circuit, and any observed eccentricity is corrected by adjustments of the screws on the sides of the universal joints. When fully adjusted the full-bridge circuit is used (see Figure 7), and the specimen is again loaded several times to 5 to 10 percent of its estimated

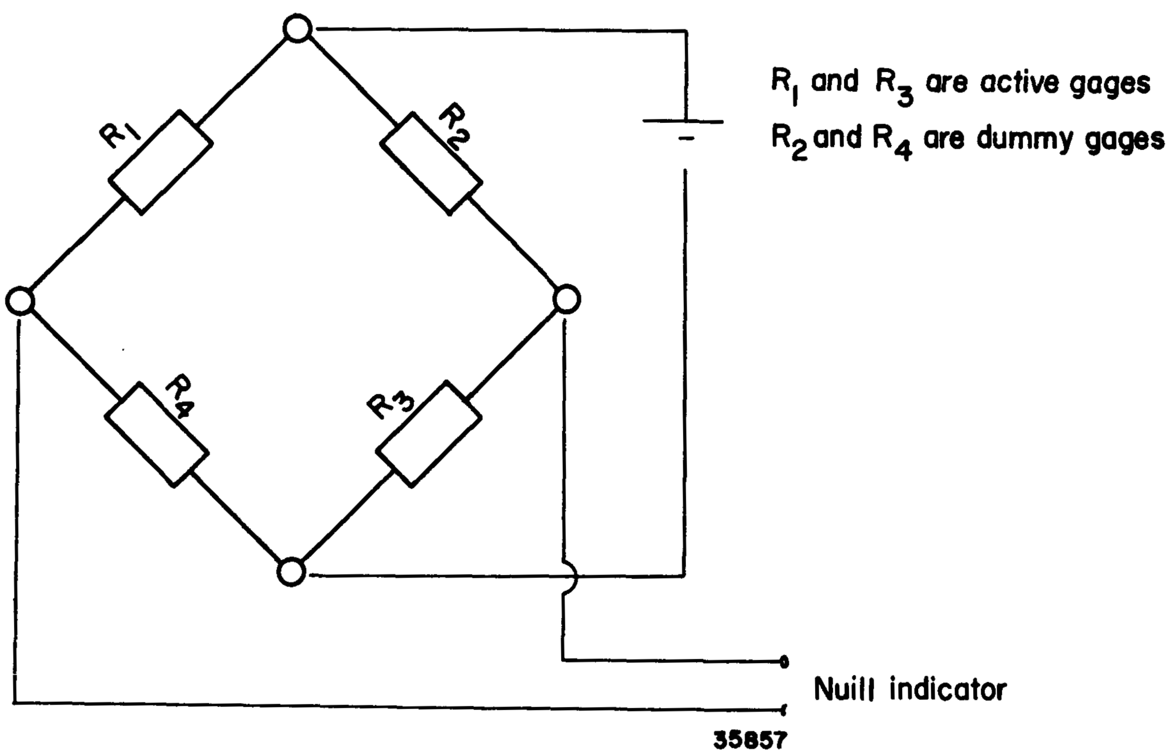

FIGURE 7. A FULL BRIDGE MEASURING CIRCUIT

MYS to assure stability in the strain readings. The load is then raised to a higher value and immediately removed. In the unloaded state, the lower pin is disengaged in the slot so that only a nominal load (~1/2 lb) remains on the specimen. At this reference load, the strain is read. This procedure is repeated, with the load increasing incrementally with each cycle, until a total permanent elongation of the order of 50 microinches per inch is read under the reference load.

At the conclusion of the MYS test, the specimen was normally loaded incrementally, and strain readings were taken at each stress level. From these data, elastic moduli were calculated.

EXPERIMENTAL RESULTS

A typical example of the data generated is given in Figure 8. There is normally a stress range over which no measurable plastic deformation occurs. Once deformation begins, however, the curve begins to break over sharply. The load represented by the intersection of the curve and the vertical line represents the microyield stress at one microinch per inch (MYS, 1 μin./in.). It is also significant to measure the microyield stress at five and 10 microinches per inch (MYS, 5 μin./in. and MYS, 10 μin./in.) as these give some indication of strain hardening and may relate to creep resistance. Data of this type are given in Table 4.

Although it is premature to draw binding conclusions on relatively sparse data, it is nevertheless interesting to point out several pertinent characteristics of these data. These are:

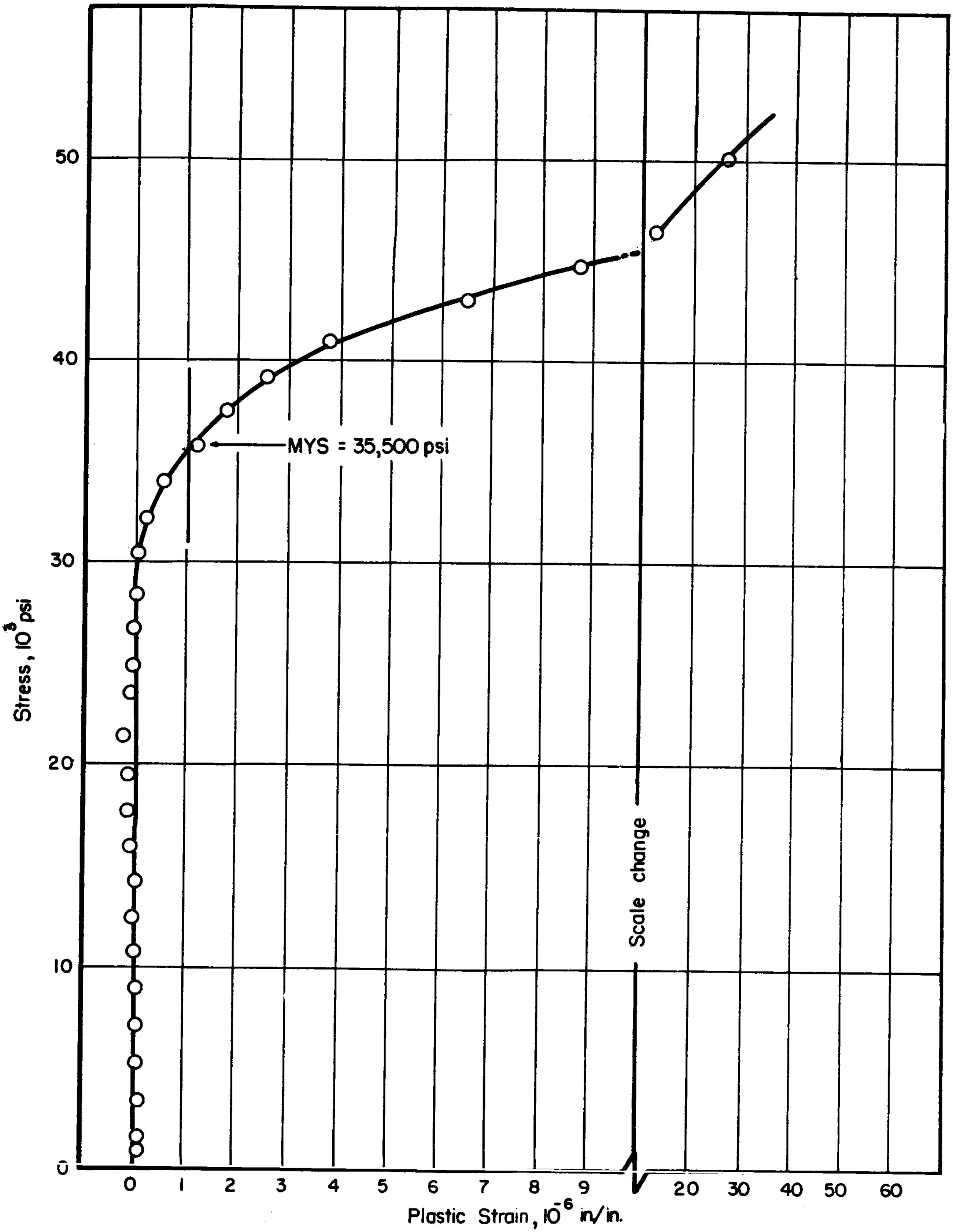

FIGURE 8. 2024-T4 ALUMINUM, SPECIMEN NUMBER TWO, 400 F FOR ONE HOUR

TABLE 4. PRECISION MEACHANICAL PROPERTIES OF VARIOUS MATERIALS

Material	Specimen Number	Heat Treatment	MYS, 1 μin./in. psi	MYS, 5 μin./in. psi	MYS, 10 μin./in. psi
2024-T4 Al	8	As received	38,000	45,000	46,200
	7	As received	39,000	42,500	44,500
	2	1 hr at 400 F	35,500	42,000	45,500
	3	1 hr at 400 F	29,000	39,500	44,500
	19	1 hr at 450 F	28,800	37,000	41,200
	18	1 hr at 450 F	32,400	39,000	43,000
	21	1 hr at 500 F	20,200	28,200	32,200
	20	1 hr at 500 F	22,500	30,000	33,500
5456-H34 Al	4	As received	17,500	19,800	--
	5	As received	17,500	19,400	20,900
	10	1 hr at 400 F	21,600	22,300	22,800
	11	1 hr at 400 F	19,500	21,000	21,600
	17	1 hr at 450 F	22,200	24,800	25,600
	16	1 hr at 450 F	19,800	21,000	21,700
	22	1 hr at 500 F	19,700	20,800	21,000
	23	1 hr at 500 F	18,600	20,000	20,400
6061-T6 Al	2	As received	19,000	26,400	30,400
	1	As received	18,400	26,800	30,400
	9	1 hr at 400 F	25,400	29,900	31,600
	8	1 hr at 400 F	27,200	31,600	33,900
	15	1 hr at 450 F	20,800	25,000	27,100
	14	1 hr at 450 F	21,400	26,400	28,400
	25	1 hr at 500 F	10,200	16,200	19,200
	24	1 hr at 500 F	17,200	19,900	22,000
AZ 31 Mg	1	As received	3,200	4,400	4,800
	2	As received	3,200	4,200	4,700
	3	1 hr at 450 F	3,340	3,880	4,080
	4	1 hr at 450 F	3,850	5,240	5,840
TZM Mo	1	As received	24,000	52,000	61,000
	2	As received	35,500	58,000	65,000
	4	1 hr at 2200 F in H_2	52,200	60,500	63,000
	5	1 hr at 2200 F in H_2	52,700	62,500	67,000
	6	1 hr at 2200 F in H_2	52,700	61,000	67,000
	7	1 hr at 2200 F in H_2	58,000	63,500	67,500
	8	1 hr at 2600 F in H_2	49,700	54,000	61,500

TABLE 4. (Continued)

Material	Specimen Number	Heat Treatment	MYS, 1 μin./in. psi	MYS, 5 μin./in. psi	MYS, 10 μin./in. psi
TZM Mo	9	1 hr at 2600 F in H_2	50,000	53,000	56,000
	10	1 hr at 2600 F in H_2	49,000	52,000	54,000
	11	1 hr at 2600 F in H_2	52,000	56,500	58,000
I-400 Be	1	As received	7,400	14,200	20,000
	2	As received	5,000	10,400	15,500
	6	1 hr at 1100 F in vacuum	9,000	18,600	24,000
	7	1 hr at 1100 F in vacuum	6,800	17,000	24,000
	4	1 hr at 1500 F in vacuum	9,200	17,600	22,700
	5	1 hr at 1500 F in vacuum	5,200	12,100	16,800

(1) There is a significant scatter in the experimental results between what are nominally identical specimens. For example, the MYS, 1 μin./in. values for identical I-400 Be samples differed by 40 percent or more in all three modifications tested. There is little doubt that these variations are far beyond the experimental errors involved in making the measurements. The implication is that there are some uncontrolled factors involved. These may be material inhomogeneity, processing variations, surface variations, or even more subtle differences. The existence of such variations should suggest extreme caution in the use of such data until a sufficient number of results exist for a formal statistical analysis.

(2) In several of the alloys, namely 5456-H34 Al, 6061-T6 Al, and TZM molybdenum, there is an appreciable increase in MYS, 1 μin./in. after a moderate heat treatment. It is probably significant that at higher strains (MYS, 10 μin./in.) this increase is small or nonexistent. This serves to emphasize the point that precision mechanical properties can be strongly influenced by factors which do not alter the more conventional mechanical properties.

(3) In neither the TZM molybdenum nor the I-400 Be did recrystallization heat treatment lower the MYS, 1 μin./in. significantly below that for a lower temperature heat treatment. However, the MYS, 10 μin./in. appeared to be lower in both cases. This suggests that, in materials which are not highly alloyed, and which do not depend upon phase transformations or massive precipitation for their strength properties full recrystallization (and consequently much more effective stress relief) may be an acceptable path to take. Probably the grain size must be kept small, so such treatments should be at the lowest practical temperatures and for the shortest practical times. These data also serve to demonstrate that the softening that would be seen in conventional mechanical-property data is not necessarily to be expected in MYS, 1 μin./in. values.

(Data for this report may be found in Battelle Laboratory Record Book No. 23706, pages 1 through 82.)